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TITLE: Low alpha ray tin manufacture for electronic components - involves removing lead contained in tin alloy by refining and reducing alpha ray amount
PATENT-ASSIGNEE: MITSUBISHI MATERIALS CORP[MITV]
PRIORITY-DATA: 1997JP-0236883 (September 2, 1997)
PATENT-FAMILY:

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APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO	APPL-DATE
JP11080852A	N/A	1997JP-0236883	September 2, 1997

INT-CL_(IPC): B23K035/40; C22B025/08 ; C23C014/34
ABSTRACTED-PUB-NO: JP11080852A
BASIC-ABSTRACT: NOVELTY - An alloy of tin and lead has 10 cph/cm2 of alpha ray amount. The alloy is refined and lead content is removed so that alpha ray amount is as low as 0.0005 cph/cm2.

USE - For electronic components manufacture, for use of tin as sputtering target or CVD material.

ADVANTAGE - Tin with low alpha ray amount is manufactured effectively.
CHOSEN-DRAWING: Dwg.0/0
DERWENT-CLASS: L03 M23 M26 P55
CPI-CODES: L03-H; M23-E; M26-B04; M26-B05;

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TIX:
Low alpha ray tin manufacture for electronic components - involves removing lead contained in tin alloy by refining and reducing alpha ray amount

ABTX:
NOVELTY - An alloy of tin and lead has 10 cph/cm2 of alpha ray amount. The alloy is refined and lead content is removed so that alpha ray amount is as low as 0.0005 cph/cm2.

ABTX:
ADVANTAGE - Tin with low alpha ray amount is manufactured effectively.

TTX:
LOW ALPHA RAY TIN MANUFACTURE ELECTRONIC COMPONENT REMOVE LEAD CONTAIN TIN
ALLOY REFINE REDUCE ALPHA RAY AMOUNT

PATENT ABSTRACTS OF JAPAN

(11)Publication number : 11-080852

(43)Date of publication of application : 26.03.1999

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B23K 35/40
// C23C 14/34

(21)Application number : 09-236883

(22)Date of filing : 02.09.1997

(71)Applicant : MITSUBISHI MATERIALS CORP

(72)Inventor : OOMURA TOSHIMASA
MAKI KAZUMASA

(54) PRODUCTION OF TIN WITH LOW ALPHA-RAY DOSE

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain tin minimal in a dose of alpha rays by alloying tin and lead having an alpha-ray dose not higher than a specific value and then carrying out refining for removing lead contained in tin.

SOLUTION: The dose of alpha ray in lead to be alloyed with tin is regulated to ≤10 cph/cm2. When lead having an alpha-ray dose exceeding this value is used, the dose of alpha rays resultant from tin remaining in trace quantities cannot be reduced to ≤0.001 cph/cm2. It is desirable to regulate the amount of lead to be alloyed with tin to 0.1 to 99%. After tin and such lead are alloyed, refining for removal of lead contained in tin is performed. By this method, 210Pb contained in tin can be removed, and as a result, tin minimal in the dose of alpha ray can be produced. To be concrete, tin and lead are melted in a high purity graphite crucible in a nitrogen atmosphere by means of a high frequency induction furnace to form an alloy, and this alloy is introduced into a vacuum distillation apparatus and heated to remove lead by evaporation.

LEGAL STATUS

[Date of request for examination] 31.03.2000

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[Date of requesting appeal against examiner's decision of rejection]

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CLAIMS

[Claim(s)]

[Claim 1] Tin and the amount of alpha rays are 10 cph/cm2. The manufacture method of the low alpha dosage tin characterized by performing refinement which removes the lead contained in tin after alloying the following lead.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[The technical field to which invention belongs] For this invention, the amount of alpha rays used as raw materials for manufacturing especially electronic parts, such as solder, a sputtering target, or chemical-vacuum-deposition material, is 0.0005 cph/cm2 about the manufacture method of the very few low alpha dosage tin of the amount of alpha rays. It is related with the manufacture method of the low alpha dosage tin of the following.
[0002]
[Description of the Prior Art] Generally, it is known that tin is the raw material of solder and solder is used for manufacture of semiconductor devices, such as junction of the chip in semiconductor memory, and a substrate. These semiconductor devices are in densification and the high-capacity-ized inclination increasingly in recent years, and the alpha rays emitted from solder have come to have big influence on generating of a soft error in connection with it. Therefore, what has the few amount of alpha rays is desired, and, as for the solder used for manufacture of a semiconductor device, the tin of the amount of low alpha rays is called for also about the tin used as the raw material of solder.
[0003] As a method of obtaining low alpha dosage tin, the tin which has the grace beyond (a)99.95 % of the weight is used as an anode, and it is the electrolytic solution. Sn: The sulfamic acid which hardly contains 30 - 150 g/l and radioisotope : 30 - 200 g/l is used. Cathode current density : 0.5 - 2.0 Amp/dm2, the method of electrolyzing on degree:of solution temperature15-50 degree C conditions (refer to JP,62-1478,B), (b) JIS-K In the sulfuric-acid electrolytic solution of 50 produced using the concentrated sulfuric acid which suits the specification of the special-grade-chemical sulfuric acid specified to 8951 - 280 g/l The way (refer to JP,1-283398,A) grace electrolyzes by using for an anode plate the tin which is 99.90 % of the weight or more etc. is learned.
[0004]
[Problem(s) to be Solved by the Invention] however, low enough the amount of alpha rays of the tin obtained by these methods The amount of alpha rays of the tin obtained by electrolysis of the above (a) is 0.2 cph/cm2. It is the following. The amount of alpha rays of the tin furthermore obtained by electrolysis of (b) is 0.03 cph/cm2. It is the following and, now, is the amount of alpha rays of tin 0.001 cph/cm2 There is no technology which can be lowered to below and the tin of the still much more amount of low alpha rays was called for.
[0005]
[Means for Solving the Problem] As a result of inquiring for this invention persons to get low alpha dosage tin much more than before, then, the source of alpha rays emitted from (b) usual tin Grandchild nuclear-species 210 Po of 210 Pb which is radionuclide of the Pb(s) contained as an impurity almost comes out. the tin with few contents of a certain (b) aforementioned 210 Pb The amount of alpha rays is 10 cph/cm2 to usual tin. If the obtained tin lead alloy is refined by various kinds of methods (for example, the conventional above (a) or the method of (b)) after alloying the following lead and manufacturing a tin lead alloy Alloyed Pb is not only removable, but it could also remove 210 Pb and the research result of being able to obtain low alpha dosage tin with still few amounts of alpha rays conventionally was obtained.
[0006] For this invention, it accomplishes based on this research result, and (1) tin and the amount of alpha rays are 10 cph/cm2. After alloying the following lead, it has the feature in the manufacture method of low alpha dosage tin of performing refinement which removes the lead contained in tin.
[0007] It is the amount of alpha rays of tin and the lead to alloy 10 cph/cm2 For having considered as the following, the amount of alpha rays of tin and the lead to alloy is 10 cph/cm2. When the lead to exceed is used, the amount of alpha rays by the lead which carried out ultralow-volume remains is 0.001 cph/cm2. It is [the place it becomes impossible to lower to below to tin, and] the amount of alpha rays of the lead to alloy 10 cph/cm2 It was

determined as the following.
[0008] As for the amount of tin and the lead to alloy, it is desirable in that case that it is in 0.1 - 99% of within the limits. Therefore, for this invention, (2) tin and the amount of alpha rays are 10 cph/cm2. After alloying the following lead by 0.1 - 99% of within the limits, it has the feature in the manufacture method of low alpha dosage tin of performing refinement which removes the lead contained in tin.
[0009] Tin and the amount of alpha rays are 10 cph/cm2 by this invention. By performing refinement which removes tin and the alloyed lead as a reason the tin of the amount of low alpha rays will be conventionally obtained if refinement which removes the lead contained in tin is performed after alloying the following lead 210 Pb contained in tin can be removed and it is thought that tin with the low amount of alpha rays was producible.
[0010] Although the source of alpha rays of tin is mainly 210 Pb, it is difficult to measure the amount of 210 Pb(s) directly. Generally therefore, the alpha rays emitted from 210 Pb From the place emitted in case 210 Po carries out alpha decay in process in which it collapses like 210 Pb(beta disintegration) ->210Bi(beta disintegration) ->210 Po (alpha decay) ->206 Pb and it is set to 206 Pb The amount of 210 Po in tin is correctly measured by analysis, and the amount of 210 Pb(s) contained in tin by the measured value of this amount of 210 Po is evaluated.
[0011]
[Embodiments of the Invention] Sn and the amount of surface alpha rays of marketing whose amounts of surface alpha rays are 5 cph/cm2 and purity:99.99% prepared 10 cph/cm2 and Pb of marketing it is [marketing] purity:99.99%, Sn and Pb were dissolved by the high frequency induction furnace in nitrogen atmosphere and within the high-purity-graphite crucible, and the Sn-5wt%Pb alloy was manufactured.
[0012] The example 1 aforementioned Sn-5wt%Pb alloy was put into the high-purity-graphite crucible, this was inserted in the vacuum unit, and it heated at 900 degrees C among the vacuum atmosphere of 10-3Torr for 10 hours. The uptake umbrella with which this vacuum unit was cooled with water right above the installation of a crucible is installed, Pb which evaporated is solidified within a uptake umbrella and Pb is removed. Sn which remained after cooling and in the high-purity-graphite crucible was taken out, Sn which carried out vacuum distillation of this again similarly as a raw material was rolled out in thickness of 1mm, and Sn board was produced.
[0013] 4000cm2 of this alloy board after making this Sn board pass for three years It considered as the sample and 96 timing measurements of the amount of surface alpha rays were carried out by made in **-ized Analysis pin center,large (LACS-4000M and measurement minimum:0.0005 cph/cm2). the measurement minimum of this equipment -- 0.0005 cph/cm2 it is -- the place which was not able to measure the amount of surface alpha rays with this equipment to the amount of surface alpha rays -- 0.0005 cph/cm2 It turns out that it is the following and the result was shown in Table 1.
[0014] Furthermore, this Sn board made to pass for three years : make 3g into a sample and this in the liquid which dissolved with the heat hydrochloric acid After carrying out 1dpm (decay/min) addition and adjusting 208 Po solutions to a 0.5-mol HCl solution, An argentic plate is immersed for 6 hours, an argentic plate front face is made to electrodeposit 208 Po, and it is a silicon surface-barrier type alpha-rays measuring device (it SSB(s)) about this argentic plate. Measuring-plane product: 70cm, using 2 and determination-limit:0.01 cph/cm2, the alpha ray spectrum was measured for seven days, and 210 Po was measured from the amount of marker 208 Po. The determination limit of this analytical method was 2 dpm(s)/kg, with this equipment, from the place which was not able to measure 210 Po, it turns out that it is 2 dpm(s)/less than kg, and the amount of 210 Po of a sample showed the result in Table 1.
[0015] Furthermore, this Sn board made to pass for three years was made into the sample, this was dissolved in the heat hydrochloric acid, the obtained liquid was analyzed by ICP (a plasma quantometer, determination-limit:1ppm), the amount of impurity Pb(s) was measured, and the result was shown in Table 1.
[0016] In addition, performing measurement of the amount of surface alpha rays and the amount of 210 Po by the sample after progress for after [refinement] three years It is because the phenomenon in which 210 Po which is the main radionuclide in Sn decreases by refinement, and the amount of alpha rays decreases seemingly exists. It is because amount of alpha rays and 210 Po can also measure true value if 210 Pb which is the parent nuclide of 210 Po carries out disintegration, it takes 210 Po about 2.3 years to reach radioactive equilibrium and it will measure after progress for three years.
[0017] By repeating twice putting Sn of conventional example 1 marketing into a high-purity-graphite crucible independently, inserting this in a vacuum unit, and heating at 900 degrees C among the vacuum atmosphere of 10-3Torr for 10 hours Pb which Pb was evaporated like the example 1 and evaporated is solidified within a uptake umbrella. Pb was removed, Sn which remained after cooling and in the high-purity-graphite crucible was taken out, by rolling out in thickness of 1mm, Sn board was produced, the amount of surface alpha rays, 210 Po, and the amount of impurity Pb(s) after three-year progress were measured like the example 1, and the result was shown in

Table 1.

[0018] The Sn-1wt%Pb alloy obtained by dissolving by the high frequency induction furnace within the example 2 aforementioned high-purity-graphite crucible is used as an anode. Liquid composition : Sn:30g/l. and sulfamic-acid:196 g/l cathode current density:2.0 A/dm3, Solution temperature: By electrolyzing on condition that 35 degree-C**, Pb was removed, Sn was deposited in the cathode, the amount of surface alpha rays, 210 Po, and the amount of impurity Pb(s) after three-year progress of Sn board which rolled out Sn of a cathode like the example 1, and was obtained were measured like the example 1, and the result was shown in Table 1.

[0019] Sn of conventional example 2 marketing was used as the anode, Pb is removed by electrolyzing on the same conditions as an example 2, Sn was deposited in the cathode, the amount of surface alpha rays, 210 Po, and the amount of impurity Pb(s) after three-year progress of Sn board which rolled out Sn of a cathode like the example 1, and was obtained were measured like the example 1, and the result was shown in Table 1.

[0020] As the example 3 electrolytic solution, it is JIS-K. Using the special-grade-chemical sulfuric acid and high grade pure water which are specified to 8951, the sulfuric acid of acid concentration:250 g/l was produced and the solution which added gelatin:2g and beta-naphthol:2g to this was prepared. The Sn-1wt%Pb alloy obtained by dissolving by the high frequency induction furnace within the aforementioned high-purity-graphite crucible is used as an anode using this electrolytic solution. A stainless steel board is used as a cathode. Current density:0.8 A/dm3, solution temperature:45 degree C, By electrolyzing by ******, Sn was deposited in the cathode, the amount of surface alpha rays, 210 Po, and the amount of impurity Pb(s) after three-year progress of Sn board which rolled out Sn of a cathode like the example 1, and was obtained were measured like the example 1, and the result was shown in Table 1.

[0021] Sn of conventional example 3 marketing was used as the anode, Sn was deposited in the cathode by electrolyzing on the same conditions as an example 3, the amount of surface alpha rays, 210 Po, and the amount of impurity Pb(s) after three-year progress of Sn board which rolled out Sn of a cathode like the example 1, and was obtained were measured like the example 1, and the result was shown in Table 1.

[0022]

[Table 1]

種別		試料	精錬した錫の3年経過後の測定値		
			α線量 (cph/cm²)	^{210}Po量 (dpm/Kg)	不純物Pb量 (ppm)
実施例	1	Sn−1wt%Pb合金	<0.0005	<2	3
	2		<0.0005	<2	4
	3		<0.0005	<2	2
従来例	1	市販のSn	0.71	3.5×10^{3}	1
	2		1.43	7.0×10^{3}	1
	3		0.24	1.2×10^{3}	1

[0023]

[Effect of the Invention] The amount of impurity Pb(s) contained in Sn obtained from the result shown in Table 1 by the conventional examples 1-3 which refine commercial Sn independently as it is The amount of surface alpha rays is 10 cph/cm2 to commercial Sn. In spite of being fewer than the amount of impurity Pb(s) contained in Sn obtained according to the examples 1-3 which refine the Sn-Pb alloy which alloyed the following high grades Pb it turns out that the amount of alpha rays and the amount of 210 Po of Sn which were obtained according to examples 1-3 are alike and fewer than the amount of alpha rays and the amount of 210 Po of Sn which were obtained by the conventional examples 1-3 Reduction of 210 Po which is the emitter of the alpha rays in Sn of examples 1-3 is

diluted by Pb which 210 Pb in Sn alloyed, and shows that 210Pb(s) in Sn are decreasing extremely after refinement aiming at Pb removal. As mentioned above, according to the method of this invention, the very few tin of the amount of alpha rays can be manufactured, and the effect which was excellent on industry is done so.

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TECHNICAL FIELD

[The technical field to which invention belongs] For this invention, the amount of alpha rays used as raw materials for manufacturing especially electronic parts, such as solder, a sputtering target, or chemical-vacuum-deposition material, is 0.0005 cph/cm2 about the manufacture method of the very few low alpha dosage tin of the amount of alpha rays. It is related with the manufacture method of the low alpha dosage tin of the following.

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PRIOR ART

[Description of the Prior Art] Generally, it is known that tin is the raw material of solder and solder is used for manufacture of semiconductor devices, such as junction of the chip in semiconductor memory, and a substrate. These semiconductor devices are in densification and the high-capacity-ized inclination increasingly in recent years, and the alpha rays emitted from solder have come to have big influence on generating of a soft error in connection with it. Therefore, what has the few amount of alpha rays is desired, and, as for the solder used for manufacture of a semiconductor device, the tin of the amount of low alpha rays is called for also about the tin used as the raw material of solder.

[0003] As a method of obtaining low alpha dosage tin, the tin which has the grace beyond (a)99.95 % of the weight is used as an anode, and it is the electrolytic solution. Sn: The sulfamic acid which hardly contains 30 - 150 g/l and radioisotope : 30 - 200 g/l is used. Cathode current density : 0.5 - 2.0 Amp/dm2, the method of electrolyzing on degree:of solution temperature15-50 degree C conditions (refer to JP,62-1478,B), (b) JIS-K In the sulfuric-acid electrolytic solution of 50 produced using the concentrated sulfuric acid which suits the specification of the special-grade-chemical sulfuric acid specified to 8951 - 280 g/l The way (refer to JP,1-283398,A) grace electrolyzes by using for an anode plate the tin which is 99.90 % of the weight or more etc. is learned.

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EFFECT OF THE INVENTION

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TECHNICAL PROBLEM

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MEANS

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[0008] As for the amount of tin and the lead to alloy, it is desirable in that case that it is in 0.1 - 99% of within the limits. Therefore, for this invention, (2) tin and the amount of alpha rays are 10 cph/cm2. After alloying the following lead by 0.1 - 99% of within the limits, it has the feature in the manufacture method of low alpha dosage tin of performing refinement which removes the lead contained in tin.

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[0011]

[Embodiments of the Invention] Sn and the amount of surface alpha rays of marketing whose amounts of surface alpha rays are 5 cph/cm2 and purity:99.99% prepared 10 cph/cm2 and Pb of marketing it is [marketing] purity:99.99%, Sn and Pb were dissolved by the high frequency induction furnace in nitrogen atmosphere and within the high-purity-graphite crucible, and the Sn-5wt%Pb alloy was manufactured.

[0012] The example 1 aforementioned Sn-5wt%Pb alloy was put into the high-purity-graphite crucible, this was inserted in the vacuum unit, and it heated at 900 degrees C among the vacuum atmosphere of 10-3Torr for 10 hours. The uptake umbrella with which this vacuum unit was cooled with water right above the installation of a crucible is installed, Pb which evaporated is solidified within a uptake umbrella and Pb is removed. Sn which remained after cooling and in the high-purity-graphite crucible was taken out, Sn which carried out vacuum distillation of this again similarly as a raw material was rolled out in thickness of 1mm, and Sn board was produced.

[0013] 4000cm2 of this alloy board after making this Sn board pass for three years It considered as the sample and 96 timing measurements of the amount of surface alpha rays were carried out by made in **-ized Analysis pin center,large (LACS-4000M and measurement minimum:0.0005 cph/cm2). the measurement minimum of this

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[0014] Furthermore, this Sn board made to pass for three years : make 3g into a sample and this in the liquid which dissolved with the heat hydrochloric acid After carrying out 1dpm (decay/min) addition and adjusting 208 Po solutions to a 0.5-mol HCl solution, An argentic plate is immersed for 6 hours, an argentic plate front face is made to electrodeposit 208 Po, and it is a silicon surface-barrier type alpha-rays measuring device (it SSB(s)) about this argentic plate. Measuring-plane product: 70cm, using 2 and determination-limit:0.01 cph/cm2, the alpha ray spectrum was measured for seven days, and 210 Po was measured from the amount of marker 208 Po. The determination limit of this analytical method was 2 dpm(s)/kg, with this equipment, from the place which was not able to measure 210 Po, it turns out that it is 2 dpm(s)/less than kg, and the amount of 210 Po of a sample showed the result in Table 1.

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[0017] By repeating twice putting Sn of conventional example 1 marketing into a high-purity-graphite crucible independently, inserting this in a vacuum unit, and heating at 900 degrees C among the vacuum atmosphere of 10-3Torr for 10 hours Pb which Pb was evaporated like the example 1 and evaporated is solidified within a uptake umbrella. Pb was removed, Sn which remained after cooling and in the high-purity-graphite crucible was taken out, by rolling out in thickness of 1mm, Sn board was produced, the amount of surface alpha rays, 210 Po, and the amount of impurity Pb(s) after three-year progress were measured like the example 1, and the result was shown in Table 1.

[0018] The Sn-1wt%Pb alloy obtained by dissolving by the high frequency induction furnace within the example 2 aforementioned high-purity-graphite crucible is used as an anode. Liquid composition : Sn:30g/l. and sulfamic-acid:196 g/l cathode current density:2.0 A/dm3, Solution temperature: By electrolyzing on condition that 35 degree-C**, Pb was removed, Sn was deposited in the cathode, the amount of surface alpha rays, 210 Po, and the amount of impurity Pb(s) after three-year progress of Sn board which rolled out Sn of a cathode like the example 1, and was obtained were measured like the example 1, and the result was shown in Table 1.

[0019] Sn of conventional example 2 marketing was used as the anode, Pb is removed by electrolyzing on the same conditions as an example 2, Sn was deposited in the cathode, the amount of surface alpha rays, 210 Po, and the amount of impurity Pb(s) after three-year progress of Sn board which rolled out Sn of a cathode like the example 1, and was obtained were measured like the example 1, and the result was shown in Table 1.

[0020] As the example 3 electrolytic solution, it is JIS-K. Using the special-grade-chemical sulfuric acid and high grade pure water which are specified to 8951, the sulfuric acid of acid concentration:250 g/l was produced and the solution which added gelatin:2g and beta-naphthol:2g to this was prepared. The Sn-1wt%Pb alloy obtained by dissolving by the high frequency induction furnace within the aforementioned high-purity-graphite crucible is used as an anode using this electrolytic solution. A stainless steel board is used as a cathode. Current density:0.8 A/dm3, solution temperature:45 degree C, By electrolyzing by ******, Sn was deposited in the cathode, the amount of surface alpha rays, 210 Po, and the amount of impurity Pb(s) after three-year progress of Sn board which rolled out Sn of a cathode like the example 1, and was obtained were measured like the example 1, and the result was shown in Table 1.

[0021] Sn of conventional example 3 marketing was used as the anode, Sn was deposited in the cathode by electrolyzing on the same conditions as an example 3, the amount of surface alpha rays, 210 Po, and the amount of impurity Pb(s) after three-year progress of Sn board which rolled out Sn of a cathode like the example 1, and was obtained were measured like the example 1, and the result was shown in Table 1.

[0022]

[Table 1]

種別		試料	精錬した錫の３年経過後の測定値		
			α線量 (cph/cm²)	^{210}Po量 (dpm/Kg)	不純物Pb量 (ppm)
実施例	1	Sn−1wt%Pb合金	＜0.0005	＜2	3
	2		＜0.0005	＜2	4
	3		＜0.0005	＜2	2
従来例	1	市販のSn	0.71	3.5×10^{3}	1
	2		1.43	7.0×10^{3}	1
	3		0.24	1.2×10^{3}	1

[Translation done.]